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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Group Art Unit 1752

OFFICIAL

In re application of : April 9, 2003
Wenjie Li et al. : Examiner: Barbara Lee Gilliam
Serial No. : 09/886,428 :
Filed: June 21, 2001 : IBM Corporation
Title: RESIST COMPOSITIONS WITH : Dept. 18G/Bldg, 300-482
POLYMERS HAVING 2-CYANO : 2070 Route 52
ACRYLIC MONOMER : Hopewell Junction, NY
12533-6531

Declaration under 37 CFR 1.131

We, the undersigned, hereby declare as follows:

1. We are the joint inventors in the above identified patent application.
2. We have read and understood the patent application including claims 1-16 currently pending in the application as of April 9, 2003.
3. Prior to May 11, 2001, we conceived and reduced to practice of the idea for at least one embodiment of the invention for forming a resist composition comprising (a) an imaging polymer, and (b) a radiation sensitive acid generator, said imaging polymer comprising cyano-modified acrylic monomer units comprising an acrylic moiety with a cyano group pendant therefrom.
4. The above mentioned conception and reduction to practice is evidenced by the attached exhibits 1-3 which are invention disclosures prepared prior to May 11, 2001 to document the activity. The above mentioned conception and reduction to practice is further evidenced by the attached exhibits 4-6.
5. All the blocked out dates on exhibits 1-6 are prior to May 11, 2001.


6. On page 2 of exhibits 1-3, the benefits of using polymers containing cyano-modified acrylic monomers are disclosed on page 2, under items 1 and 2 of the main idea section.
7. Exhibit 4 shows laboratory notebook entries prior to May 11, 2001 documenting synthesis of resist polymers WJL-1 through WJL-5 containing cyano-modified acrylic monomer units comprising an acrylic moiety with a cyano group pendant therefrom.
8. Exhibit 5 shows fomulation sheet dated prior to May 11, 2001 for resist F801 containing polymer WJL-1 of Exhibit 4.
9. Exhibit 6 shows fomulation log printout indicating exposure of resist composition F801 prior to May 11, 2001.
10. We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information or belief are believed to be true; and further that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 or Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



Wenjie Li

4/9/03

Date




Pushkara Rao Varanasi

4/9/03

Date

Exhibit 1

	Disclosure FIS8.	
	Prepared for and/or by an IBM Attorney - IBM Confidential	
	Created By: Wenjie Li	Created On: 10:52:00 AM
	Last Modified By: Hilda Heinlein	Last modified On: 03:48:15 PM

Required fields are marked with the asterisk (*) and must be filled in to complete the form.

*Title of disclosure (in English)

Polymers and photoresist compositions for optical lithography

Summary

Status	Under Evaluation
Processing Location	FIS
Functional Area	ZZDKF002 ... DAVARI/Hofferon: Photoresist
Attorney/Patent Professional	Steven Capella/Fishkill/IBM
IDT Team	Wayne Moreau/Fishkill/IBM
Submitted Date	02:39:13 PM EST
Owning Division	MD
Incentive Program	
Lab	
Technology Code	101J1
PVT Score	No PVT score has been calculated.To calculate a PVT score, press the 'Calculate' button.

Inventors with a Blue Pages entry

Inventors: Wenjie Li/Fishkill/IBM, Pushkara Rao Varanasi/Fishkill/IBM

Inventor Name	Inventor	Inventor	Inventor	Inventor
> Li, Wenjie	2A3409	29/LRLA	533-4989	Varanasi, Pushkara Rao
Pushkara Rao Varanasi	N/A	N/A	N/A	N/A

> denotes primary contact

Inventors without a Blue Pages entry

Pushkara Rao Varanasi
 Serial Number : 808985 Company : IBM
 Citizen of : US
 E-Mail :
 Business Address :

Business Phone :
 Home Address :

polymers and photon compositions for optical lithography - continued

Exh. 1

IDT Selection

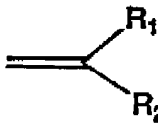
IDT Team:
Wayne Moreau/Fishkill/IBMAttorney/Patent Professional:
Steven Capella/Fishkill/IBM

Response Due to IP&L :

*Main Idea

1. Describe your invention, stating the problem solved (if appropriate), and indicating the advantages of using the invention.

A photoresist composition comprising a radiation-sensitive acid generator and a copolymer comprising a cyclic olefin as the first monomer and an olefin with two electron-withdrawing groups as the second monomer, wherein the second monomer unit has the structural formula (I):



wherein both R1 and R2 are electron-withdrawing groups such as cyano (-CN), ester (-COOR), nitro (-NO2), sulphone (-SO2R), or fluorinated alkyl group, etc. R1 and R2 could be same or different. Olefin with two electron-withdrawing groups will co-polymerize with cyclic olefins. The resulting copolymer will have good shelf-life, good mechanical properties, and good etch resistance.

2. How does the invention solve the problem or achieve an advantage, (a description of "the invention", including figures inline as appropriate)?

Maleic anhydride (MA) has to be used to form a charge-transfer complex with a cyclic olefin in order for the cyclic olefin to polymerize by free radical polymerization and/or co-polymerize with (meth)acrylate monomers. However, this system generally exhibits shelf-life issues such as undesirable thinning in the unexposed region due to MA hydrolysis. Another disadvantage is that the cyclic olefin-MA system normally has a very high glass transition temperature (Tg) due to the rigid polymer backbone, and it is not good for high baking process (annealing concept) which prevents airborne contamination of resist film and shows good PEB delay effect. Cyclic olefins are able to co-polymerize with olefins containing electron-withdrawing groups (formula (I)), eliminating the need of using MA. As a result, the copolymer has better shelf-life and more desired mechanical properties such as Tg. In the mean time, the polymer still has good etch resistance.

3. If the same advantage or problem has been identified by others (inside/outside IBM), how have those others solved it and does your solution differ and why is it better?

4. If the invention is implemented in a product or prototype, include technical details, purpose, disclosure details to others and the date of that implementation.

*Critical Questions (Questions 1-9 must be answered in English)

* Question 1

On what date was the invention workable?

Please format the date as MM/DD/YYYY

(Workable means i.e. when you know that your design will solve the problem)

Polymers and photore Compositions for optical lithography - continued

Exh. 1

* Question 2	<input type="radio"/> Yes <input checked="" type="radio"/> No
Is there any planned or actual publication or disclosure of your invention to anyone outside IBM?	
If yes, Enter the name of each publication or patent and the date published below.	
Publication/Patent: Date Published or Issued:	
Are you aware of any publications, products or patents that relate to this invention?	
<input type="radio"/> Yes <input checked="" type="radio"/> No	
If yes, Enter the name of each publication or patent and the date published below.	
Publication/Patent: Date Published or Issued:	

* Question 3	<input type="radio"/> Yes <input checked="" type="radio"/> No
Has the subject matter of the invention or a product incorporating the invention been sold, used internally in manufacturing, announced for sale, or included in a proposal?	
Is a sale, use in manufacturing, product announcement, or proposal planned?	
<input type="radio"/> Yes <input checked="" type="radio"/> No	
If Yes, identify the product if known and indicate the date or planned date of sale, announcements, or proposal and to whom the sale, announcement or proposal has been or will be made.	
Product: Version/Release: Code Name: Date: To Whom:	
If more than one, use cut and paste and append as necessary in the field provided.	

* Question 4	<input type="radio"/> Yes <input checked="" type="radio"/> No
Was the subject matter of your invention or a product incorporating your invention used in public, e.g., outside IBM or in the presence of non-IBMers?	
If yes, give a date. Please format the date as MM/DD/YYYY	

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* Question 6	<input type="radio"/> Yes <input checked="" type="radio"/> No <input type="radio"/> Not sure
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If Yes, enter the contract number	

* Question 7	<input type="radio"/> Yes <input checked="" type="radio"/> No <input type="radio"/> Not Sure
Was the invention made in the course of any alliance, joint development or other contract activities?	
If Yes, enter the following:	
Name of Alliance, Contractor or Joint Developer	

ymers and photore, compositions for optical lithography - continued

Exh. 1

Contract ID number
Relationship contact name
Relationship contact E-mail
Relationship contact phone

* Question 8	<input type="radio"/> Yes
Have you, or any of the other inventors, submitted this same invention disclosure or similar invention disclosure previously?	<input checked="" type="radio"/> No
If Yes, please provide disclosure number below:	

* Question 9	<input type="radio"/> Yes
Are you, or any of the other inventors, aware of any related inventions disclosures submitted by anyone in IBM previously?	<input checked="" type="radio"/> No
If Yes, please provide the docket or disclosure number or any other identifying information below:	

Question 10
What type of companies do you expect to compete with inventions of this type? <i>Check all that apply.</i>
<input type="checkbox"/> Manufacturers of enterprise servers
<input type="checkbox"/> Manufacturers of entry servers
<input type="checkbox"/> Manufacturers of workstations
<input type="checkbox"/> Manufacturers of PCs
<input type="checkbox"/> Non-computer manufacturers
<input type="checkbox"/> Developers of operating systems
<input type="checkbox"/> Developers of networking software
<input type="checkbox"/> Developers of application software
<input type="checkbox"/> Integrated solution providers
<input type="checkbox"/> Service providers
<input checked="" type="checkbox"/> Other (Please specify below)
photorealist manufacturers

Question 11
If the invention relates to a product or service that is outside the scope of your business unit, please recommend IBM business unit(s), IBM location(s) or individual(s) within IBM that you think would provide a good evaluation of your invention:

Patent Value Tool (Optional - this may be used by the inventor and attorney to assist with the evaluation.)
(The Patent Value tool can be used by the inventor(s) to determine the potential licensing value of your invention.)

No PVT score has been calculated. To calculate a PVT score, press the 'Calculate' button.

Market

What is the anticipated annual market size (in dollars) that will be captured by your invention?

CLAIMS

Question 1 - How new is the technical field?

Polymers and photoreactive compositions for optical lithography - continued

Exh. 1

Question 2 - How central is the invention to the product(s) which might be expected to contain the invention?

Question 3 - What is the scope of the claim?

PORTFOLIO NEED

What are the portfolio needs in the area of your invention?

EXPLOITATION & ENFORCEMENT

Question 1 - How easily can the use of the invention by a competitor be detected?

Question 2 - How easily can the use of the invention be avoided by a competitor?

BUSINESS VALUE

Question 1 - What percentage of the companies producing products in the field of this invention might use this invention?

Question 2 - What is the value of this patent to current or anticipated Alliance Activity between IBM and other companies?

Question 3 - What is the value of this patent to current or anticipated Technology Transfer Activity between IBM and other companies?

Question 4 - Does it result in prestige to IBM?

Post Disclosure Text & Drawings

Enter any additional information relating to this disclosure below:

(Form Revised 12/17/97)

Exh. 2.

**Disclosure File**

Prepared for and/or by an IBM Attorney - IBM Confidential

Created By: **Wen Li** Created On: 11:08:11 AM
 Last Modified By: **Hilda Heinlein** Last Modified On: 11:13:32 AM

Required fields are marked with the asterisk (*) and must be filled in to complete the form.

* Title of disclosure (in English)

Photoresist compositions for optical lithography

Summary

Status	Under Evaluation
Processing Location	FIS
Functional Area	ZZDKF002 ... DAVAR/Hoffman: Photoresist
Attorney/Patent Professional	Steven Capella/Fishkill/IBM
IDT Team	Wayne Moreau/Fishkill/IBM
Submitted Date	12-06-04 PM EST
Owning Division	MD
Lab	
Technology Code	101J1
PVT Score	No PVT score has been calculated. To calculate a PVT score, press the 'Calculate' button.

Inventors with a Blue Pages entry

Inventors: **Wen Li**/Fishkill/IBM, **Pushkara Rao** Varanasi/Fishkill/IBM

Inventor Name	Inventor Serial	Div/Dept	Inventor Phone	Manager Name
> W. Li	2A3409	29/LRLA	533-4989	Varanasi, Pushkara Rao

> denotes primary contact

Inventors without a Blue Pages entry

Pushkara Rao Varanasi

Serial Number : 808985

Citizen of : US

E-Mail :

Business Address :

Company : IBM

Business Phone :

Home Address :

IDT Selection

3 Photoresist comp ons for optical lithography - continued

Exh-2

IDT Team:

Wayne Moreau/Fishkill/IBM

Attorney/Patent Professional:

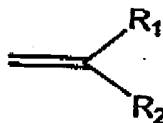
Steven Capella/Fishkill/IBM

Response Due to IP&L :

*Main Idea

1. Describe your invention, stating the problem solved (if appropriate), and indicating the advantages of using the invention.

A photoresist composition comprising a radiation-sensitive acid generator and a copolymer comprising a cyclic olefin as the first monomer and an olefin with two electron-withdrawing groups and at least one of them also contains an acid-labile group as the second monomer, wherein the second monomer unit has the structural formula (I):



, wherein both R1 and R2 are electron-withdrawing groups such as cyano (-CN), ester (-COOR), nitro (-NO2), sulphone (-SO2R), or fluorinated alkyl group, ect. R1, R2, or both R1 and R2 also contains an acid-labile group. R1 and R2 could be same or different. Olefin with two electron-withdrawing groups will co-polymerize with cyclic olefins. The resulting copolymer will have good shelf-life, good mechanical properties, and good etch resistance.

2. How does the invention solve the problem or achieve an advantage, (a description of "the invention", including figures inline as appropriate)?

Maleic anhydride (MA) has to be used to form a charge-transfer complex with a cyclic olefin in order for the cyclic olefin to polymerize by free radical polymerization and/or co-polymerize with (meth)acrylate monomers. However, this system generally exhibits shelf-life issues such as undesirable thinning in the unexposed region due to MA hydrolysis. Another disadvantage is that the cyclic olefin-MA system normally has a very high glass transition temperature (Tg) due to the rigid polymer backbone, and it is not good for high baking process (annealing concept) which prevents airborne contamination of resist film and shows good PEB delay effect. Cyclic olefins are able to co-polymerize with olefins containing electron-withdrawing groups (formula (I)), eliminating the need of using MA. As a result, the copolymer has better shelf-life and more desired mechanical properties such as Tg. In the mean time, the polymer still has good etch resistance.

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4. If the invention is implemented in a product or prototype, include technical details, purpose, disclosure details to others and the date of that implementation.

No

*Critical Questions (Questions 1-9 must be answered in English)

*Question 1

On what date was the invention workable?

Please format the date as MM/DD/YYYY

(Workable means i.e. when you know that your design will solve the problem)

*Question 2

*resist compositions for optical lithography - continued

Exh. 2

Is there any planned or actual publication or disclosure of your invention to anyone outside IBM?	<input type="radio"/> Yes <input checked="" type="radio"/> No
If yes, Enter the name of each publication or patent and the date published below.	
Publication/Patent:	
Date Published or Issued:	
Are you aware of any publications, products or patents that relate to this invention?	<input type="radio"/> Yes <input checked="" type="radio"/> No
If yes, Enter the name of each publication or patent and the date published below.	
Publication/Patent:	
Date Published or Issued:	

*Question 3	<input type="radio"/> Yes <input checked="" type="radio"/> No
Has the subject matter of the invention or a product incorporating the invention been sold, used internally in manufacturing, announced for sale, or included in a proposal?	
Is a sale, use in manufacturing, product announcement, or proposal planned?	<input type="radio"/> Yes <input checked="" type="radio"/> No
If Yes, identify the product if known and indicate the date or planned date of sale, announcements, or proposal and to whom the sale, announcement or proposal has been or will be made.	
Product:	
Version/Releaser:	
Code Name:	
Date:	
To Whom:	
If more than one, use cut and paste and append as necessary in the field provided.	

*Question 4	<input type="radio"/> Yes <input checked="" type="radio"/> No
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Was the invention, in any way, started or developed under a government contract or project?	
If Yes, enter the contract number	

*Question 7	<input type="radio"/> Yes <input checked="" type="radio"/> No <input type="radio"/> Not Sure
Was the invention made in the course of any alliance, joint development or other contract activities?	
If Yes, enter the following:	
Name of Alliance, Contractor or Joint Developer	

photoresist compositions for optical lithography - continued

Exh. 2

Contract ID number
Relationship contact name
Relationship contact E-mail
Relationship contact phone

*** Question 8**

Have you, or any of the other inventors, submitted this same invention disclosure or similar invention disclosure previously? ☐ Yes
☒ No

If Yes, please provide disclosure number below:

*** Question 9**

Are you, or any of the other inventors, aware of any related inventions disclosures submitted by anyone in IBM previously? ☐ Yes
☒ No

If Yes, please provide the docket or disclosure number or any other identifying information below:

Question 10

What type of companies do you expect to compete with inventions of this type? Check all that apply.

- ☐ Manufacturers of enterprise servers
- ☐ Manufacturers of entry servers
- ☐ Manufacturers of workstations
- ☐ Manufacturers of PC's
- ☐ Non-computer manufacturers
- ☐ Developers of operating systems
- ☐ Developers of networking software
- ☐ Developers of application software
- ☐ Integrated solution providers
- ☐ Service providers
- ☒ Other (Please specify below)
Photoresist manufacturers

Question 11

If the invention relates to a product or service that is outside the scope of your business unit, please recommend IBM business unit(s), IBM location(s) or individual(s) within IBM that you think would provide a good evaluation of your invention:

Patent Value Tool (Optional - this may be used by the inventor and attorney to assist with the evaluation of your invention.)
(The Patent Value tool can be used by the inventor(s) to determine the potential licensing value of your invention.)

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CLAIMS

Question 1 - How new is the technical field?

Question 2 - How central is the invention to the product(s) which might be expected to contain the

Photoresist compositions for optical lithography - continued

Exh. 2-

invention?

Question 3 - What is the scope of the claim?

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Question 4 - Does it result in prestige to IBM?

Post Disclosure Text & Drawings

Enter any additional information relating to this disclosure below:

(Form Revised 12/17/97)

Exh 3

	Disclosure FIS8	
	Prepared for and/or by an IBM Attorney, IBM Confidential	
	Created By: Wayne Li	Created On: 11:57:38 AM
	Last Modified By: Hilda Heinlein	Last Modified On: 11:18:35 AM

Required fields are marked with the asterisk (*) and must be filled in to complete the form.

*Title of disclosure (in English)
Polymers and photoresist compositions for optical lithography

Summary

Status	Under Evaluation
Processing Location	FIS
Functional Area	ZZDKF002 ... DAVARI/Helfferon: Photoresist
Attorney/Patent Professional	Steven Capella/Fishkill/IBM
IDT Team	Wayne Moreau/Fishkill/IBM
Submitted Date	12:17:55 PM EST
Owning Division	MD
Lab	
Technology Code	101J1
PVT Score	No PVT score has been calculated. To calculate a PVT score, press the 'Calculate' button.

Inventors with a Blue Pages entry

Inventors: **Wayne Li/Fishkill/IBM**

Inventor Name	Inventor Serial	Inventor Dept	Inventor Phone	Manager Name
> Li, Wayne	2A3409	291RLA	533-4989	Varanasi, Pushkara Rao

> denotes primary contact

Inventors without a Blue Pages entry

Pushkara Rao Varanasi

Serial Number : 808985

Citizen of : US

E-Mail :

Business Address :

Company : IBM

Business Phone :

Home Address :

IDT Selection

Polymers and photoresist compositions for optical lithography - continued

Exh. 3

IDT Team:

Wayne Moreau/Fishkill/IBM

Attorney/Patent Professional:

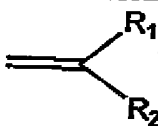
Steven Capella/Fishkill/IBM

Response Due to IP&L :

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wherein both R1 and R2 are electron-withdrawing groups such as cyano (-CN), ester (-COOR), nitro (-NO2), sulphone (-SO2R), or fluorinated alkyl group, ect. R1, R2, or both R1 and R2 also contains a polarity-modifying group such as lactone, amide, or a polar group having a heteroatom with an electronegativity greater than 3.00, ect. R1 and R2 could be same or different. Olefin with two electron-withdrawing groups will co-polymerize with cyclic olefins. The resulting copolymer will have good shelf-life, good mechanical properties, and good etch resistance.

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No

***Critical Questions (Questions 1-9 must be answered in English)**

***Question 1**

On what date was the invention workable?

Please format the date as MM/DD/YYYY

(Workable means i.e. when you know that your design will solve the problem)

Polymers and photoresist compositions for optical lithography - continued

Exh. 3

*** Question 2**

Is there any planned or actual publication or disclosure of your invention to anyone outside IBM? ☐ Yes ☒ No

If yes, Enter the name of each publication or patent and the date published below.

Publication/Patent:

Date Published or Issued:

Are you aware of any publications, products or patents that relate to this invention? ☐ Yes ☒ No

If yes, Enter the name of each publication or patent and the date published below.

Publication/Patent:

Date Published or Issued:

*** Question 3**

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If Yes, identify the product if known and indicate the date or planned date of sale, announcements, or proposal and to whom the sale, announcement or proposal has been or will be made.

Product:

Version/Release:

Code Name:

Date:

To Whom:

If more than one, use cut and paste and append as necessary in the field provided.

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*** Question 6**

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If Yes, enter the contract number ☐ Not sure

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If Yes, enter the following: ☐ Not Sure

Name of Alliance, Contractor or Joint Developer

layers and photoresist compositions for optical lithography - continued

Exh. 3

Contract ID number
Relationship contact name
Relationship contact E-mail
Relationship contact phone

*** Question 8**

Have you, or any of the other inventors, submitted this same invention disclosure or similar invention disclosure previously? ☐ Yes ☒ No

If Yes, please provide disclosure number below:

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- ☐ Manufacturers of workstations
- ☐ Manufacturers of PC's
- ☐ Non-computer manufacturers
- ☐ Developers of operating systems
- ☐ Developers of networking software
- ☐ Developers of application software
- ☐ Integrated solution providers
- ☐ Service providers
- ☒ Other (Please specify below)
photoresist manufacturers

Question 11

If the invention relates to a product or service that is outside the scope of your business unit, please recommend IBM business unit(s), IBM location(s) or individual(s) within IBM that you think would provide a good evaluation of your invention:

Patent Value Tool (Optional - this may be used by the inventor and attorney to assist with the evaluation of your invention.)
(The Patent Value tool can be used by the inventor(s) to determine the potential licensing value of your invention.)

Market

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CLAIMS

Question 1 - How new is the technical field?

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ers and photorests compositions for optical lithography - continued

Exh.3

invention?

Question 3 - What is the scope of the claim?

PORTFOLIO NEED

What are the portfolio needs in the area of your invention?

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Question 4 - Does it result in prestige to IBM?

Post Disclosure Text & Drawings

Enter any additional information relating to this disclosure below:

(Form Revised 12/17/97)

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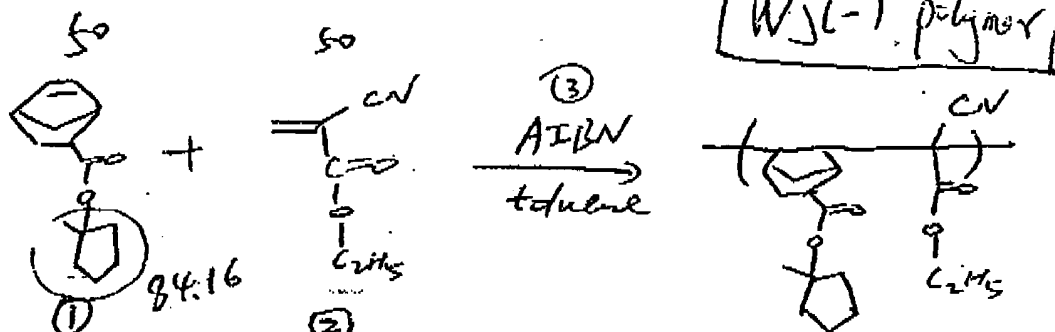
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EXH. 4

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IBM Technical Notebook



	M.W.	Weight	Mole
①	220.3	8.8g	0.004
②	125.1	5g (4.92g)	0.004
③	164	n/135mg	0.04 x 2%

toluene. 50ml (43.7g). (24% solid content).

Glass bottle and starting material (d.o.b. 3/74) was heated in oven at 130°C for 2 days. The bottle was charged with ③ and ① and some anhydrous toluene. The bottle was sealed and bubbled with Ar for 1h. ② and the bottle was transferred to dry box. ② was added to the solution. The bottle was taken out of the dry box and was heated on a oil bath to 100°C slowly over 0.5h. The mixture was (oil temp.).

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Exh. 4

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Stirred at 80-90°C for an additional hour. As the temp was raised to above 70°C, bubbles was observed from the solution. As rxn went on, some ~~gel~~ (from decar of AIBN) particles precipitated out. The mixture was precipitated into ~350-400 mL hexanes. The remaining gel particles were found not soluble in THF and stable in CH_3NO_2 , ~~THF~~ and were separated aside. The precipitate was collected by filtration and washed with hexanes (50 mL x 2) and further with 50 mL CH_3OH (with 3 drops of HCl added) and dried at 60°C in vacuum oven overnight. ~4.9 g white solid was collected.

yield: ↓
36%

^{13}C NMR showed it is the copolymer ~40% NB-MCP incorporated.

TGA showed 20% weight loss at 220°C, corresponding to MCP deprotection (~~40%~~ composition) based on

The polymer was found to be soluble in THF, acetone, cyclohexanone, *p*-butyrolactone.

not so soluble in PMA.

$$\overline{M}_n = 6472$$

$$\overline{M}_w = 12233$$

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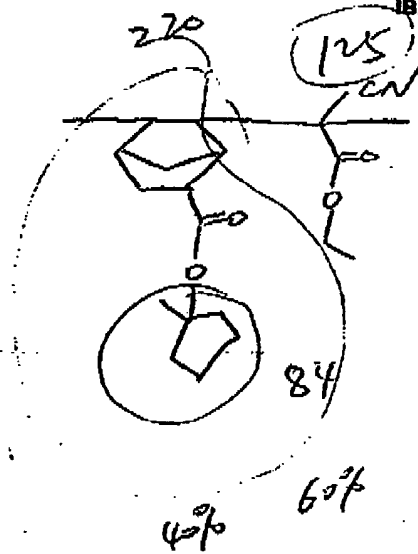
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$$220 \times 40\% + 125 \times 60\% = 163$$

Mcp deprotection weight loss:

$$84 \times 40\% / 163 = 20.6\%$$

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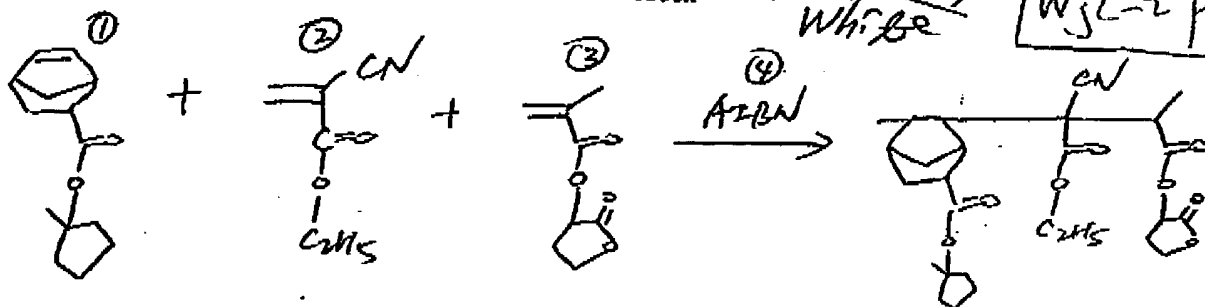
Exh. 4

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(bar)
White

WJL-2 polymer



40

40

20

	M.w.	weight	mole
①	220.3	8.81	0.04
②	125.1	5 (5.2)	0.04
③	170.2	3.4 (3.47)	0.02
④	164	0.33 (0.33)	0.1 x 2%

toluene

60 ml (52.4g) — 25% solution

Same procedure as on p.20. As the temperature was brought to -70°C , the polymerization occurred right away. The solution was stirred at -70 – -80°C (oil temperature) for 10 min. Gel-like polymer precipitated out and stuck to the bottom of the bottle. Magnetic stirring bar was not able to stir anymore. The bottle was shaken with hands ^{occasionally} and put in the oil bath for another 20 min. The solution was cooled to -70°C and precipitated into 400 ml Hexanes, the gel-like polymer was dissolved in ^{more} THF + 5 ml acetone and precipitated into hexanes too. The solids was washed with 5 ml ethyl and dried ~~under~~ under vacuum at 60°C overnight.

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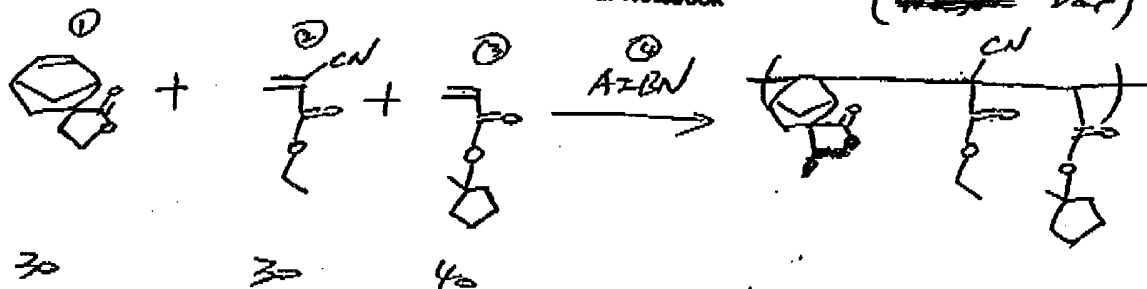
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	M.W.	weight	mole
①	164.2	4.93	0.03
②	125.1	3.75	0.03
③	154.2	6.17	0.04
④	164	0.328 (2.31)	0.1 x 2%

toluene 60 ml

gel formation while adding ②, could be due to moisture in ①.

WJL-2 polymer 1: gel-like portion precipitated into CH₃OH - 1.48g
 WJL-2 polymer 2: precipitate from hexanes - 1.58g
 13C NMR showed both are the same.
 11% MCPNB 52% ECNA 37% GBLMA
 yield 19%

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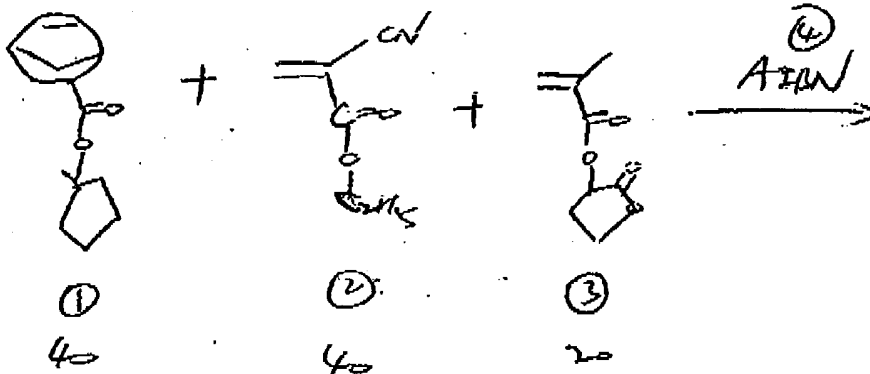
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Exh. 4

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W3L-3 polymer

	M.W.	weight	mole
①	220.3	7.05 (7.18)	0.032
②	125.1	4.0 (4.3)	0.032
③	172.2	2.72 (2.88)	0.016
④	164	~135 mg (150 mg)	0.08 x 10

toluene

60 ml + 15 ml = 75 ml (66g) → 18%

Same procedure as p20. The rxn was carried out in a 250ml RB flask instead of bottle → better stirring efficiency. Also, more solvent was used. ~~the~~ As the T was brought above 70°C (oil temp.) polymerization started right away. Gel-like polymer was stuck to the flask. The solution was stirred for another 0.5h. The polymer was precipitated into ~450ml Hexanes.

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Exh. 4

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The gel-like polymer stuck to the flask was dissolved in THF (25ml) + acetone (ca 3ml), and precipitated into hexanes too. The combined polymer was washed with 50ml CH_3OH (with 3 drops of HCl) and dried under vacuum at 60°C overnight. ~ 2.4g white solid was obtained.

↓
17% yield.

^{13}C NMR showed again only some amount of MCP-NB was incorporated into the polymer backbone.

$$\overline{M}_n = 30095$$

$$\overline{M}_w = 59404$$

$$P_d = 1.97.$$

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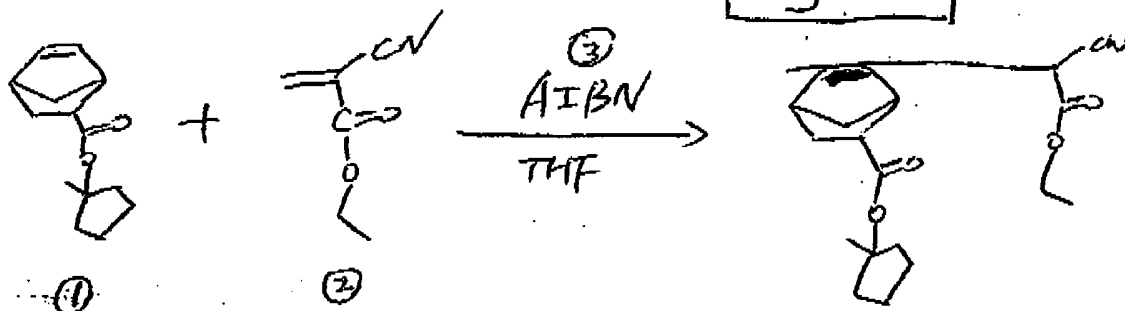
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Exh. 4

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WJL-5



	M.W.	weight	mole
①	220.3	8.8	0.04
②	125.1	5	0.04
③	164	0.262	0.008125

THF (d 0.89) 62ml \rightarrow 20% ~~monomer~~ homopolymer
 anhydrous
 ①+② + 62ml THF was bubbled with Ar for 50 min
 ② was added in dropwise dry box. The resulting solution
 was heated to $\sim 75^\circ\text{C}$ (oil bath) slowly over 40 min,
 and then was stirred at that temp. overnight.
 The solution was cooled to r.t. and (in 22 h) precipitated into $\sim 450\text{ml}$
 Hexanes. The white precipitate was collected by filtration
 and washed with Hexanes (50ml x 1) and methanol ($\sim 30\text{ml}$).

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Exh. 4

IBM Technical Notebook

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and dried under vacuum at 60°C overnight.
~5.6g white solid was obtained.
Yield 41%.

The solid was further dissolved in THF and precipitated into MeOH.

~3.2g white solid was obtained.

$\overline{M}_w = 5113$ GPC evaluation:

$\overline{M}_n = 4089$ clean profiles.

$Pd = 1.25$

thickness 1.55!!

~1.9g white solid was obtained after concentrating the MeOH filtrate. $\rightarrow \overline{M}_w = 2333$

$\overline{M}_n = 1615$

$Pd = 1.44$

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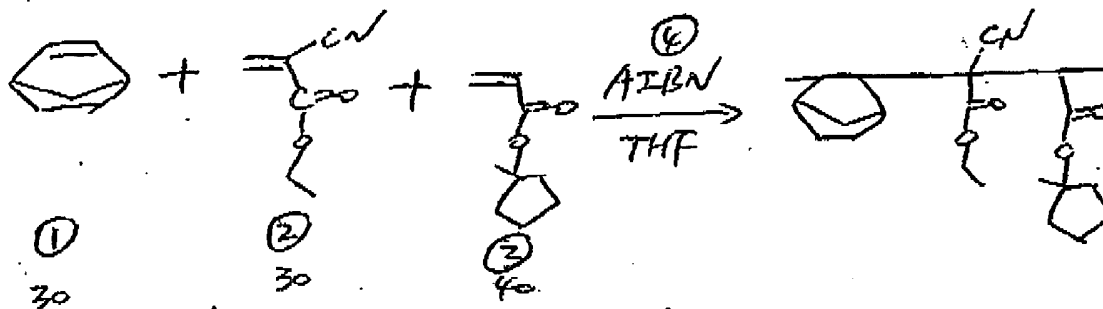
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	M.W.	Weight	mole
①	94	3.4	0.03 x 1.2
②	125.1	3.75	0.03
③	154.2	6.2	0.04
④	164	0.33	0.1 x 0.02

THF 61ml → 20% monomer

The above understood

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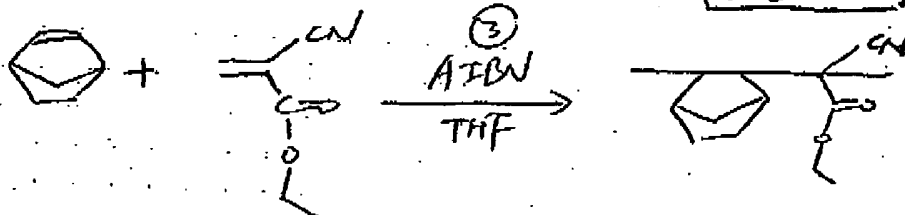
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Exh. 4

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WJL-4



	M.W.	weight	mele
①	94	1.41	0.015
②	125.1	1.88	0.015
③	164	0.1	0.03 x 2p

THF

18ml → 20% monomer

Same procedure as p28. 1/50ml Hexanes was used for precipitation. The white ~~solid~~ precipitate was collected by filtration and dried under vacuum at 60°C overnight. 2.2g white solid was obtained.
 Yield 67%.

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Phase 3 mixed PAGs & solution - (P1>0, if P1=0 next page)
F801

Exh. 5

Design Properties		Design	Actual	% wt solid
Total %solid		11		
(p1+p2)/polymer (wt%)		3.5		
PFOS/polymer (wt %) - P1				
P1/P2				
P2/polymer (wt %)				
(p1+p2)/base (molar ratio)		0.500	ERR	
Base/polymer (wt %)		10.052		
D.I./polymer (%)	Bock			
Solvent A (%)	BAD			
Solvent B (%)	PMA	100		
surfactant/solution (ppm)		0		
Poly solution A loading (g)		0		h

Formulation #

F801

Components	Description	%solid in solvent	plan loading (g)	actual wt (g)
Polymer solution A	WJL-1 polymer			
Polymer solution B	in solution		10.0000	10.005
PAG 1 Lot 42099	HPFOS	100	0.0000	
PAG 2 Lot 102199	MDPFBS		0.0450	44.5mg
Base in solvent A	Bock		0.0075	7.7mg
B-AD lot 120899	BAD		0.3000	0.3004
Solvent A	PMA cyclohexane	0	0.0000	
Solvent B		0	3.7884	3.7453g
surfactant		0	0.0000	
		5	0.0000	

Total Solution (g)

14.1409

Note: loading order

- 1) polymer
- 2) solvent A & B
- 3) roll until polymer resolve
- 4) charge PAG1 & 2 & roll
- 5) charge base & surfactant

Note: Remove 31.57 gm at the end and add 7.5 mg I-OAC

$E = 22.7\%$

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Exh. 6

EO ID	SAMPLE	THICKNESS	Litho Spin Spd	PAB	PEB	DEV	nk/pl
LB5A			1800	130°/60"	130°/90"	AD10 60"	
LBXA			2080	"	"	"	6.3
LC0A			1900	"	"	"	6.1
LENA			1728	"	"	"	6.1
LFPA	F801		2000	"	"	"	5.7
			3280	"	190°/60"	"	22

Exposer: 193 Nikon Stepper 0.6NA

IBM UL 3

225°/120"

5KÅ @ 2399 rpm

AR19

215°/90"

~620Å

Sample ID	Litho Wafer ID	Nom Dose (mJ)	Incr (mJ)	Focus (μ)	Pattern	Illumination sigma
AR165J-FA8	LBVA	20.8	0.83	-0.1±0.15		2/3
AR165J-FA9	LBVA	14.4	0.6	"		"
AR165J-FA10	LBWA	17.6	0.7	"		"
AR237	LBVA	18.2	0.76	"		"
R60-2/5k UL-3	LHEA	23.6	0.95	0±0.1	BONE CN FULL T4	0.75
R60-2/ARCH	QNTA	"	"	"	BONE CN M1MC T4	"
AT5000J-ACT35	LB2A	"	"	"	"	"
AT5000J-ACT37	RBNA	37.5	1.5	"	"	"
AT5000J-ACT38	KBPA	38.5	1.5	"	"	2/3
	KBMA	"	"	"	"	"

EO start dose for F801 1mJ in 0.5 mJ increments
EO start dose for all other wafers is 1.5 in 0.2mJ increments